

AD-A091 792

# TECHNICAL LIBRARY

AD

TECHNICAL REPORT ARLCB-TR-80037

## DETERMINATION OF PHASE TRANSFORMATION TEMPERATURES OF TITANIUM-NICKEL USING DIFFERENTIAL THERMAL ANALYSIS

R. Vincent Milligan

October 1980



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND  
LARGE CALIBER WEAPON SYSTEMS LABORATORY  
BENÉ WEAPONS LABORATORY  
WATERVLIET, N. Y. 12189

AMCMS No. 611102H420011

DA Project No. 1L161102AH42

PRON No. 1A0217141A1A

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

#### DISCLAIMER

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

The use of trade name(s) and/or manufacturer(s) does not constitute an official indorsement or approval.

#### DISPOSITION

Destroy this report when it is no longer needed. Do not return it to the originator.

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER ARLCB-TR-80037	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) DETERMINATION OF PHASE TRANSFORMATION TEMPERATURES OF TITANIUM-NICKEL USING DIFFERENTIAL THERMAL ANALYSIS		5. TYPE OF REPORT & PERIOD COVERED
7. AUTHOR(s) R. Vincent Milligan		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Benet Weapons Laboratory Watervliet Arsenal, Watervliet, NY 12189 DRDAR-LCB-TL		8. CONTRACT OR GRANT NUMBER(s)
11. CONTROLLING OFFICE NAME AND ADDRESS US Army Armament Research and Development Command Large Caliber Weapon Systems Laboratory Dover, NJ 07801		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS AMCMS No. 611102H420011 DA Project No. 1L161102AH42 PRON No. 1A0217141A1A
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE October 1980
		13. NUMBER OF PAGES 18
		15. SECURITY CLASS. (of this report)  UNCLASSIFIED
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited.		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES  Published in Proceedings of Fourth International Conference on Titanium, Kyoto, Japan, 19-22 May 1980.		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Nickel Titanium Nickel-Titanium Differential Thermal Analysis Phase Transformations		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Some of the more popular methods used to determine phase transformations in metals are x-ray, dilatometry, and electrical resistivity. Data reported for the TiNi alloy using Differential Thermal Analysis (DTA) is quite sparse and it appears that little effort has been made to correlate these results with x-ray, dilatometry, or resistivity data. The purpose of this investigation was to determine the $M_s$ and $A_s$ temperatures for several alloys having compositions (CONT'D ON REVERSE)		

## 20. Abstract (Cont'd)

near 50 atomic percent titanium. The DTA method was used. The results are compared with those reported by several investigators that used different techniques. The DTA data obtained shows excellent agreement with Kornilov's  $A_s$  temperatures,<sup>1</sup> as a function of composition, obtained by dilatometry. A small variation was found between the results of this study and Wasilewski's x-ray data,<sup>2</sup> and Hanlon's resistivity measurements<sup>3</sup> for the  $M_s$  temperatures. It is concluded from this investigation that DTA analysis is a credible method for determining phase transformation temperatures for the TiNi material.

## TABLE OF CONTENTS

	<u>Page</u>
ACKNOWLEDGMENT	ii
INTRODUCTION	1
EXPERIMENTAL MATERIALS USED	3
RESULTS AND DISCUSSION	4
CONCLUSIONS	5
REFERENCES	6

## TABLES

I. VARIOUS METHODS USED TO STUDY PHASE TRANSFORMATIONS	2
--------------------------------------------------------	---

## LIST OF ILLUSTRATIONS

1. Change in Differential Temperature vs Temperature.	8
2. $M_s$ Temperature vs. Atomic Percent Titanium.	9
3. $M_s$ Temperature vs. Atomic Percent Titanium.	10
4. $M_s$ Temperature vs. Atomic Percent Titanium.	11
5. $M_s$ Temperature vs. Atomic Percent Titanium.	12
6. $A_s$ Temperature vs. Atomic Percent Titanium.	13
7. $A_s$ Temperature vs. Atomic Percent Titanium.	14
8. $A_s$ Temperature vs. Atomic Percent Titanium.	15
9. $A_s$ Temperature vs. Atomic Percent Titanium.	16

#### ACKNOWLEDGMENT

I would like to express appreciation for the help received from Fred Stimler of Goodyear Aerospace and Paul Cote of Benet Weapons Laboratory, LCWSL in carrying out these experiments.

#### NOTE

Manufacturers and products mentioned herein do not constitute an official endorsement or approval by the Department of the Army.

## INTRODUCTION

Many different methods have been used to determine phase transformation temperatures in metals. Table I lists some of the more popular ones - though not necessarily in their order of importance. References cited in the tables are examples and not meant to be all inclusive. For the most part, the investigators listed studied the titanium-nickel (TiNi) system, but there are some exceptions. Many additional references are listed in references 10 and 11. The selected references cited in the text are not in the same order as those listed on the reference page.

Although there have been some efforts using Differential Thermal Analysis,<sup>1,4,12</sup> the data is quite sparse and it appears that little effort has been made to correlate the results with other techniques for the TiNi system. The objective of the study was to determine transformation temperatures using Differential Thermal Analysis (DTA) and then compare these results with those obtained by x-ray, dilatometry, and electrical resistivity methods.

---

References are listed at the end of this report.

TABLE I. VARIOUS METHODS USED TO STUDY PHASE TRANSFORMATIONS

Author	Method	Ref. No.
R.J. Wasilewski et al	X-Ray	1
I.I. Kornilov et al	Dilatometry	2
W.B. Cross et al	Elect. Resistivity	3
H.U. Schuerch	Diff. Thermal Anal.	4
G.R. Speich et al	Acoustic Emission	5
R.R. Hasiguti et al	Internal Friction	6
N.G. Pace et al	Ultrasonic	7
C.M. Wayman	Metallographic Techs.	8
K.H. Eckelmeyer	Strain-Temp Techs.	9



## EXPERIMENTAL MATERIALS USED

Three different heats of the material were supplied by Titanium Metals Corp. and some additional material by Reactive Metals, Inc. in the form of one-half inch diameter rods. These alloys had compositions in the range of 49 to 51 atomic percent titanium. Thin slices 1/16 inch thick were cut from the rods using a low speed metallurgical saw. Small pie-shaped pieces weighing approximately 15 to 25 milligrams were then cut out of the disks so that they would fit into the pan of the Differential Thermal Analyzer.

Some material was kindly supplied by Goodyear Aerospace Corp. in the form of 100 mil diameter wire. This was cut into thin disks and tested in the DTA without further cutting.

The equipment used was a Dupont 990 Differential Thermal Analyzer. The heating and cooling rates were 10°C per minute. Upon cooling, an exothermal peak was obtained which was indicative of the  $M_s$  temperature. The  $M_s$  temperature is defined as the temperature at which the parent phase starts to transform to Martensite. On subsequent heating an endothermic peak was obtained and gave the  $A_s$  temperature. The  $A_s$  temperature is defined as the temperature at which the martensite starts to transform back to the parent phase. For specimens having rather low  $M_s$  temperatures, a cold cell filled with liquid  $N_2$  was used to provide adequate cooling. Figure 1 is a copy of an actual trace showing these peaks.

## RESULTS AND DISCUSSION

Figure 2 is a replot of an  $M_s$  transformation curve using data obtained by Kornilov et al.<sup>2</sup> by the method of dilatometry. Here I have taken the liberty to plot the transformation temperatures vs. atomic percent titanium rather than atomic percent nickel as done by Kornilov. For brevity, Figure 2 also contains a plot of x-ray data from Table I of reference 1 as well as a replot of resistivity data obtained from Figure 7 by Hanlon et al.<sup>13</sup> In this case I have used °C on the ordinate rather than °K used by the authors. In Figure 3,  $M_s$  data obtained in the present study by the DTA method is presented. Figure 4 shows some miscellaneous DTA data superimposed on the line from Figure 3 along with some additional resistivity data by Cross et al.<sup>3</sup> With the exception of the one data point by Wang et al.,<sup>12</sup> the data agrees quite well with that obtained in the present study. Figure 5 is a summary plot showing a superposition of the results for the  $M_s$  temperatures from Figures 2 to 4.

Results from the present study are about as far to the right of the Wasilewski and Hanlon lines as the Kornilov results are to the left. The overall difference in the results in the temperature range from -60° to +60° is less than one percent on the composition axis. An average for the four studies would be close to the Wasilewski-Hanlon curves. The very striking thing about these results is how sensitive the transition temperature is to composition.

Figure 6 shows a replot of Kornilov's data for the  $A_s$  temperatures. Also included are some data from Figure 3 of Eckelmeyer's report.<sup>9</sup> Eckelmeyer obtained this data by straining the specimens approximately four percent, unloading, heating, and then observing the temperature at which the specimen

starts to contract (revert back) to its original length. Unfortunately, for this comparison, most of the data is located in the composition range where the transformation temperature is relatively insensitive to temperature. Figure 7 is a plot of data obtained in the present study by the DTA method. Figure 8 is a plot of some miscellaneous DTA data as well as some resistivity data superimposed on the results from Figure 7. In this case the scatter seems to be a little larger than in the plot for the  $M_s$  temperatures. Finally, Figure 9 is a superposition of the results from Figures 6 and 7 showing excellent agreement for the three separate studies.

Results from this study indicate that DTA can be a viable method for determining phase transformation temperatures in the TiNi system. If carefully done, it can provide information less expensively than that obtained by using highly sophisticated equipment such as x-ray. It could be a helpful ancillary tool in stress-assisted martensitic, shape memory transformation studies.

## CONCLUSIONS

1. Based on the excellent agreement of the data from this study with that previously obtained by other methods, it can be concluded that DTA is a credible method for determining phase transformation temperatures in the TiNi system.
2. The sensitivity of the  $M_s$  and  $A_s$  temperatures with composition which is of the order of  $100^\circ\text{C}$  for one atomic percent increase in titanium indicates the potential problems that can arise in seeking repeatability of behavior for TiNi material.

#### REFERENCES

1. R. J. Wasilewski, S. R. Butler, and J. E. Hanlon, "On the Martensitic Transformation in TiNi," Jour. of Met. Sci., Vol. 1, 1967, pp. 104-110.
2. I. I. Kornilov, Ye V. Kachur, and O. K. Belousov, "Diffraction Analysis of Transformation in the Compound TiNi," Fiz. Met. Metalloved, Vol. 32, No. 2, 1971, pp. 420-422.
3. W. B. Cross, A. H. Kariotis, and F. J. Stimler, "Nitinol Characterization Study," NASA CR 1433, September 1969.
4. H. U. Schuerch, "Certain Physical Properties and Applications of Nitinol," NASA CR 1232, November 1969.
5. G. R. Speich and R. M. Fisher, "Acoustic Emission During Martensite Formation," ASTM STP 505, May 1972, pp. 140-151.
6. R. R. Hasiguti and K. Iwasaki, "Correlation Between Plastic Deformation and Phase Changes in the Compound TiNi With Special Reference to Internal Friction," Supp. to Trans. Jap. Inst. of Metals, Vol. 9, 1968, pp. 288-291.
7. N. G. Pace and G. A. Saunders, "Ultrasonic Study of the Martensitic Phase Change in TiNi," Phil. Mag., Vol. 22, No. 175, July 1970, pp. 73-82.
8. C. M. Wayman, "Deformation, Mechanisms, and Other Characteristics of Shape Memory Alloys," Shape Memory Effects in Alloys, Plenum Press, J. Perkins, Ed., May 1975, pp. 1-27.
9. K. H. Eckelmeyer, "The Effect of Alloying on Shape Memory Phenomenon in Nitinol," Scripta Met., Vol. 10, 1976, pp. 667-672.

10. C. M. Jackson, H. J. Wagner, and R. J. Wasilewski, "55-Nitinol - The Alloy With a Memory: Its Physical Metallurgy, Properties, and Applications," NASA SP 5110, 1972.
11. Shape Memory Effects in Alloys, Plenum Press, J. Perkins, Ed., May 1975.
12. F. E. Wang, B. F. DeSavage, and W. J. Buehler, "The Irreversible Critical Range in the TiNi Transition," Jour. of Applied Physics, Vol. 39, No. 5, April 1968, pp. 2166-2175.
13. J. E. Hanlon, S. R. Butler, and R. J. Wasilewski, "Effect of Martensitic Transformation on the Electrical and Magnetic Properties of TiNi," Trans. AIME, Vol. 239, September 1967, pp. 1323-1327.

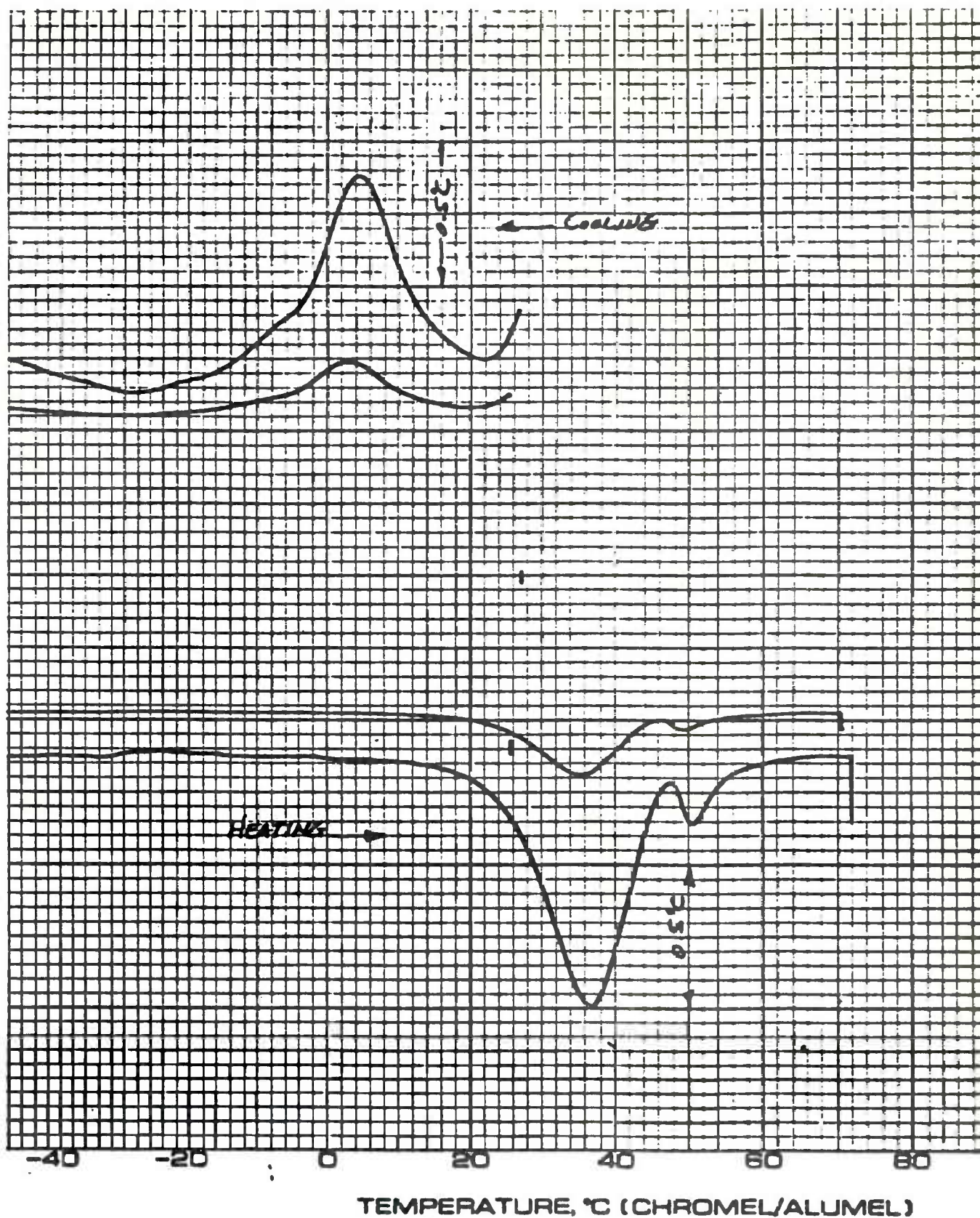


Fig 1. Change in Differential Temperature vs Temperature. Traces were obtained using a Differential Thermal Analyzer. The top trace shows an exothermic peak on cooling and the bottom trace shows an endothermic peak on subsequent heating. The smaller peaks are the same data on another pen with a suppressed vertical scale.



Figure 2

$M_s$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM

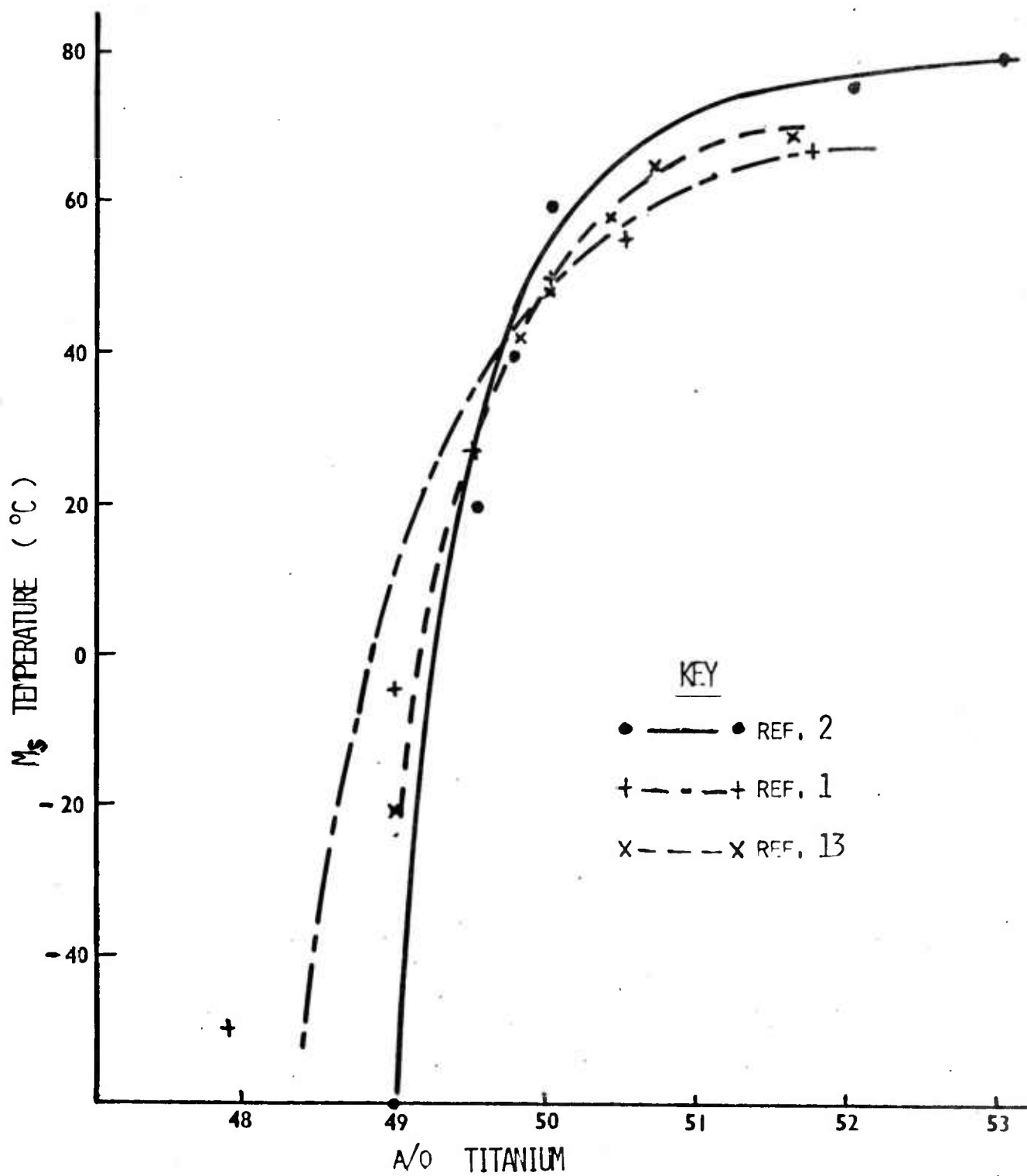


Figure 3

$M_S$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM

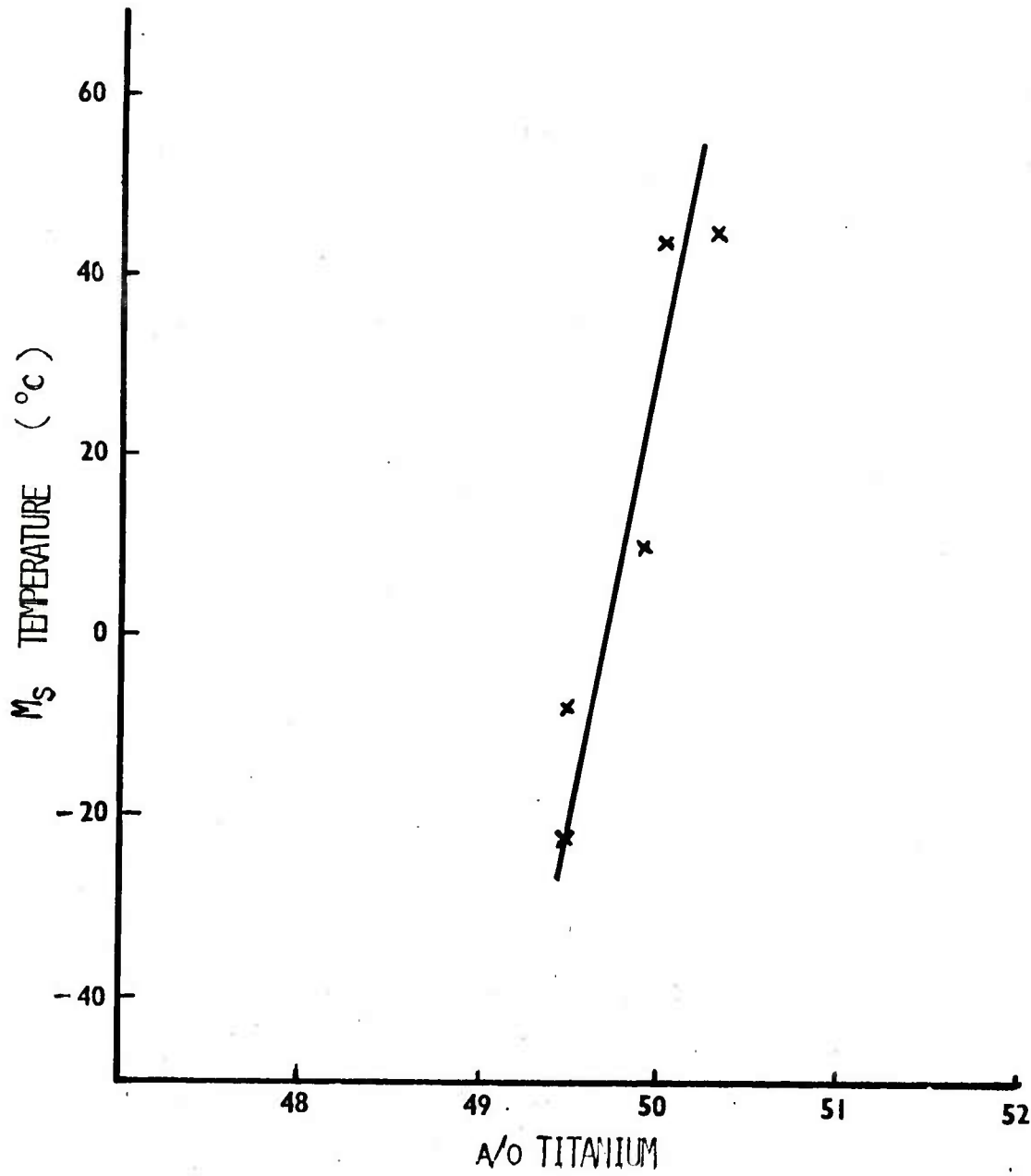




Figure 4  
 $M_s$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM

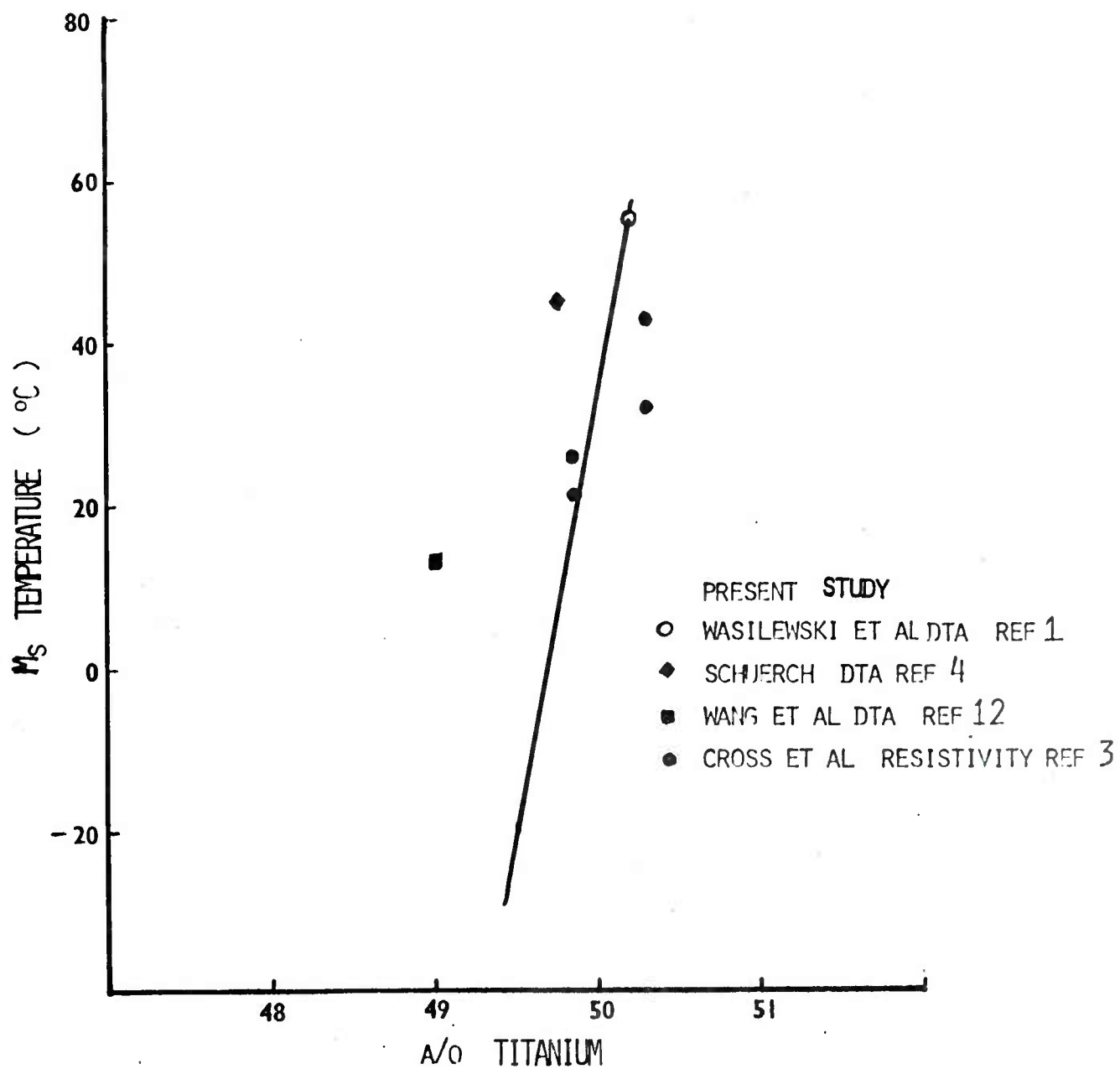


Figure 5

$M_s$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM

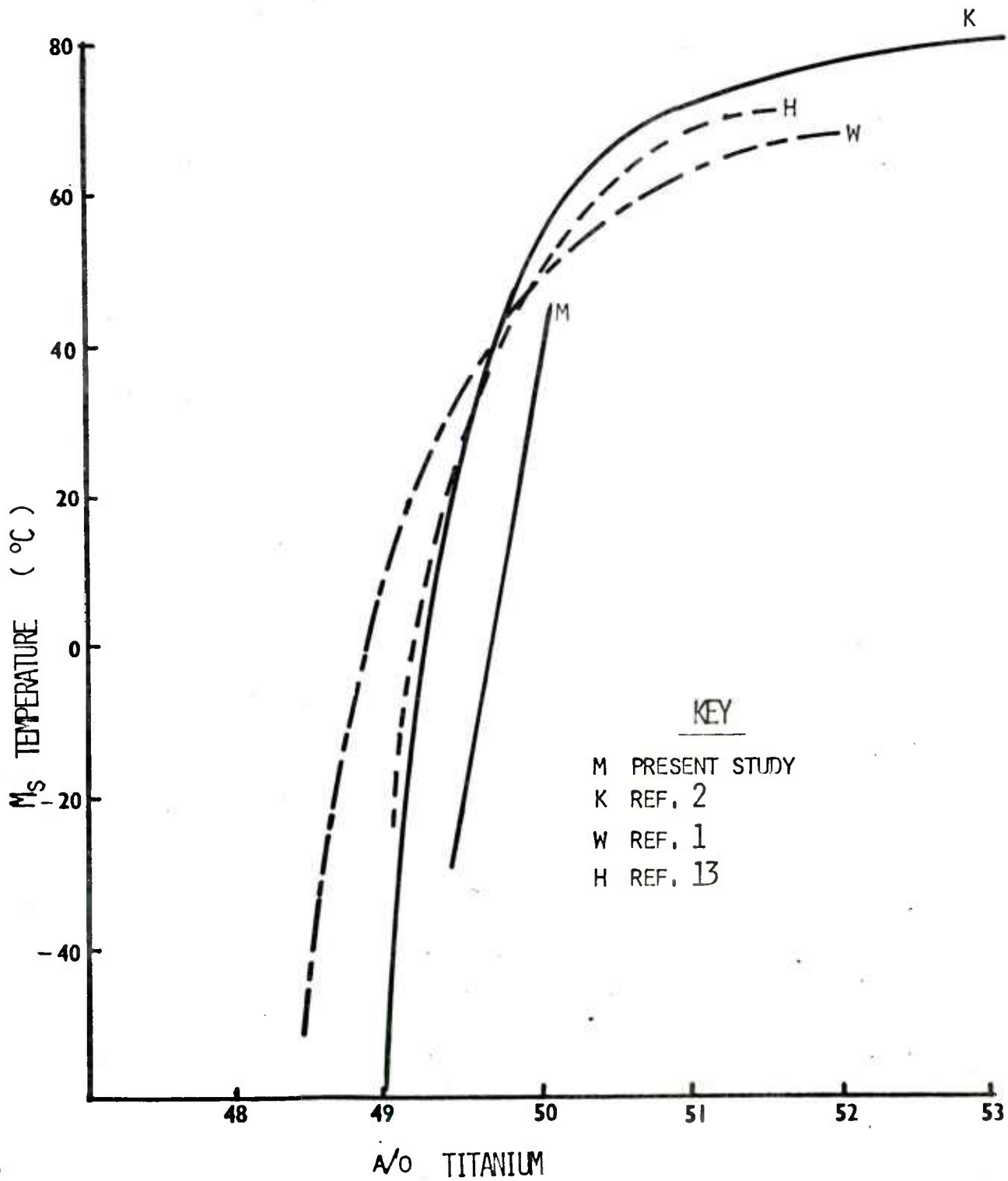


Figure 6

$A_S$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM

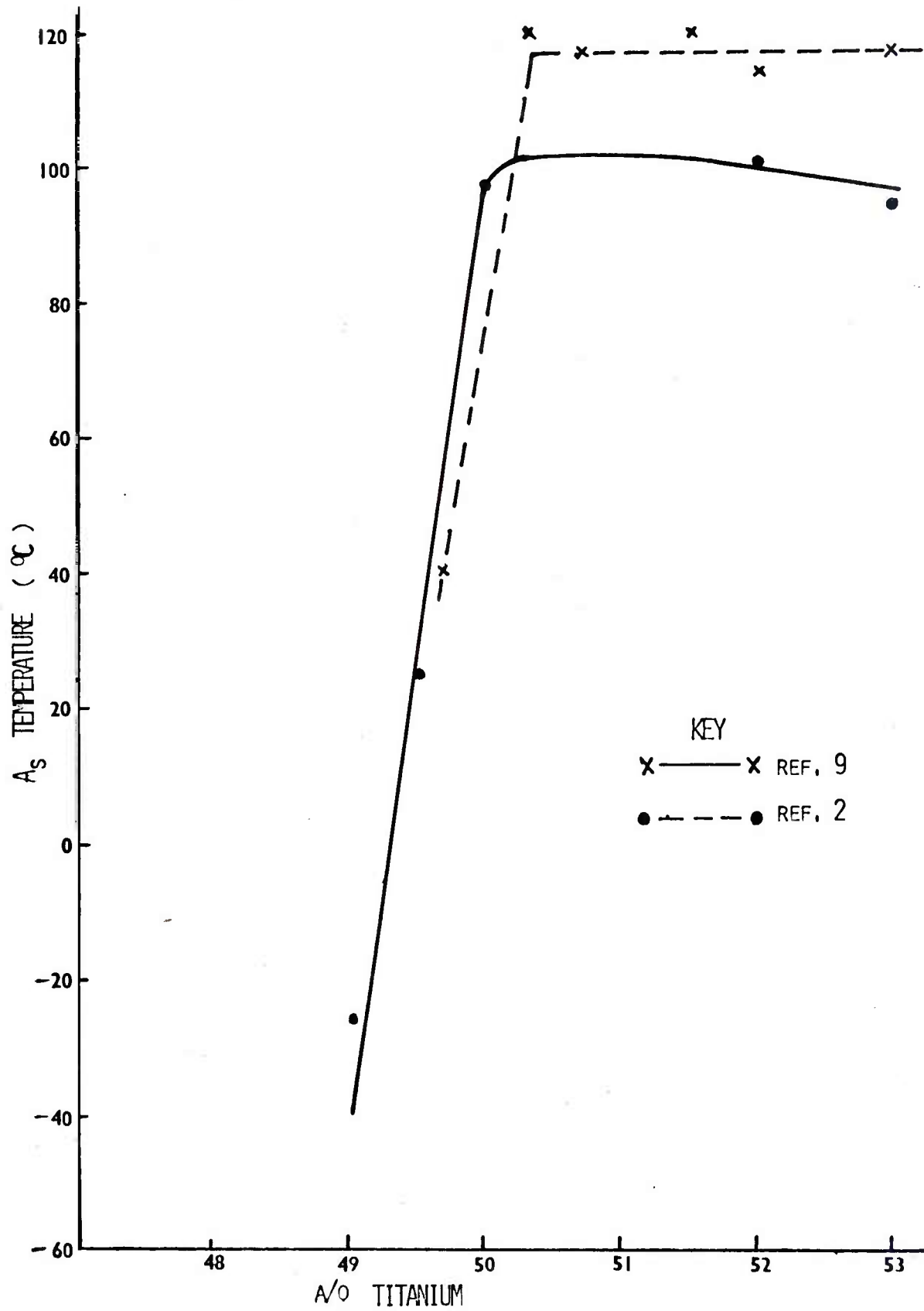


Figure 7

$A_S$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM

PRESENT DTA STUDY

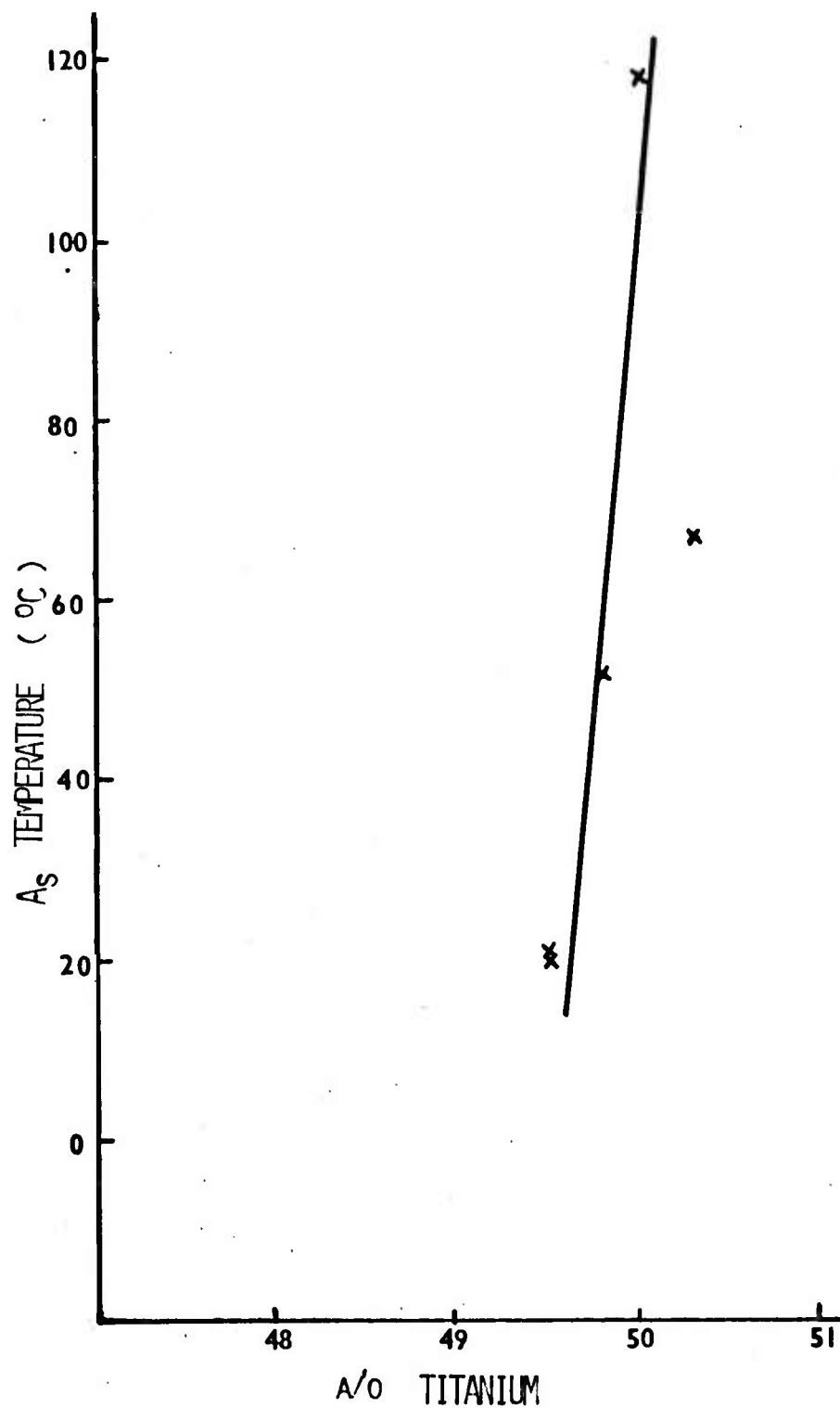


Figure 8

$A_S$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM

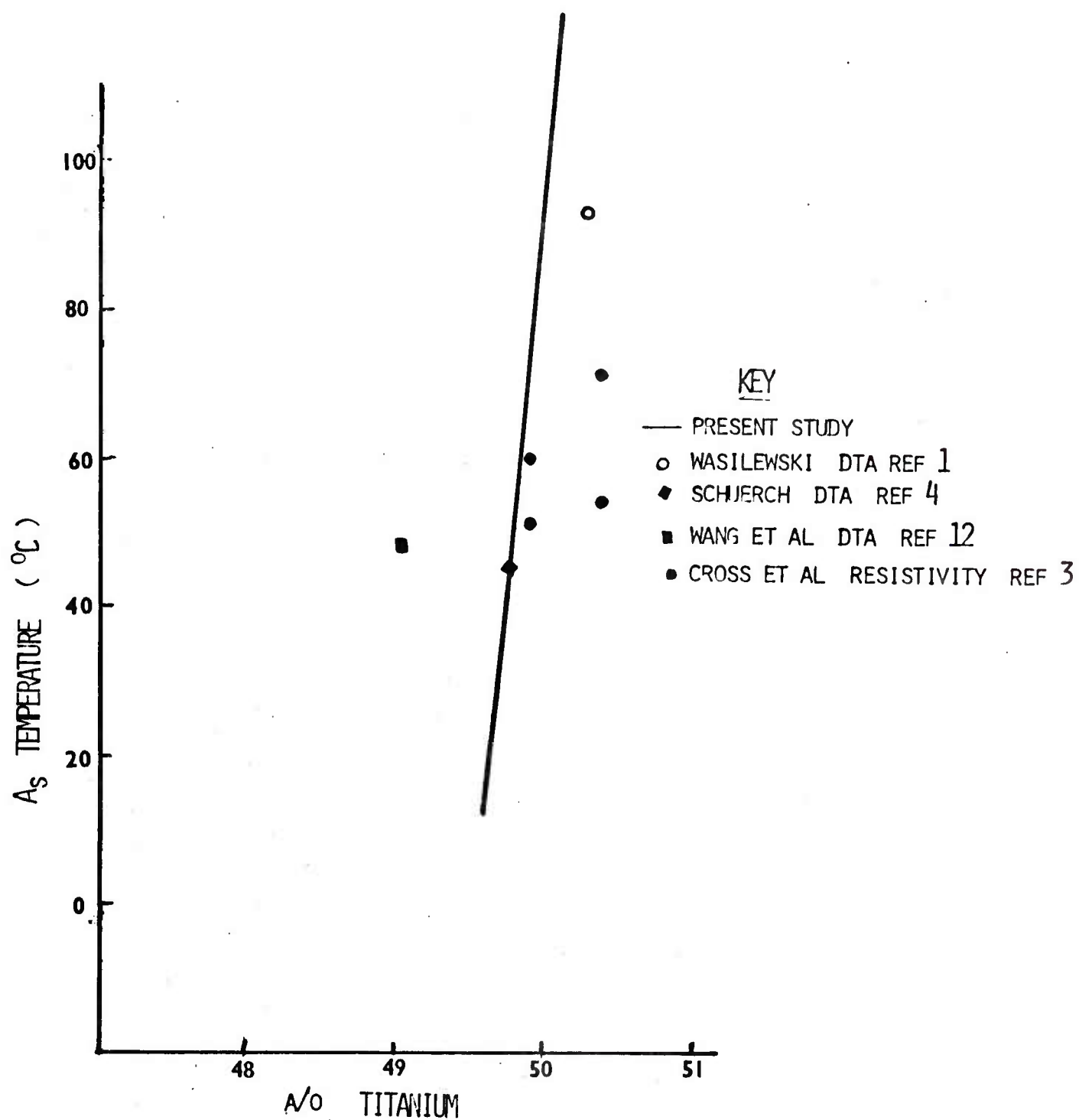
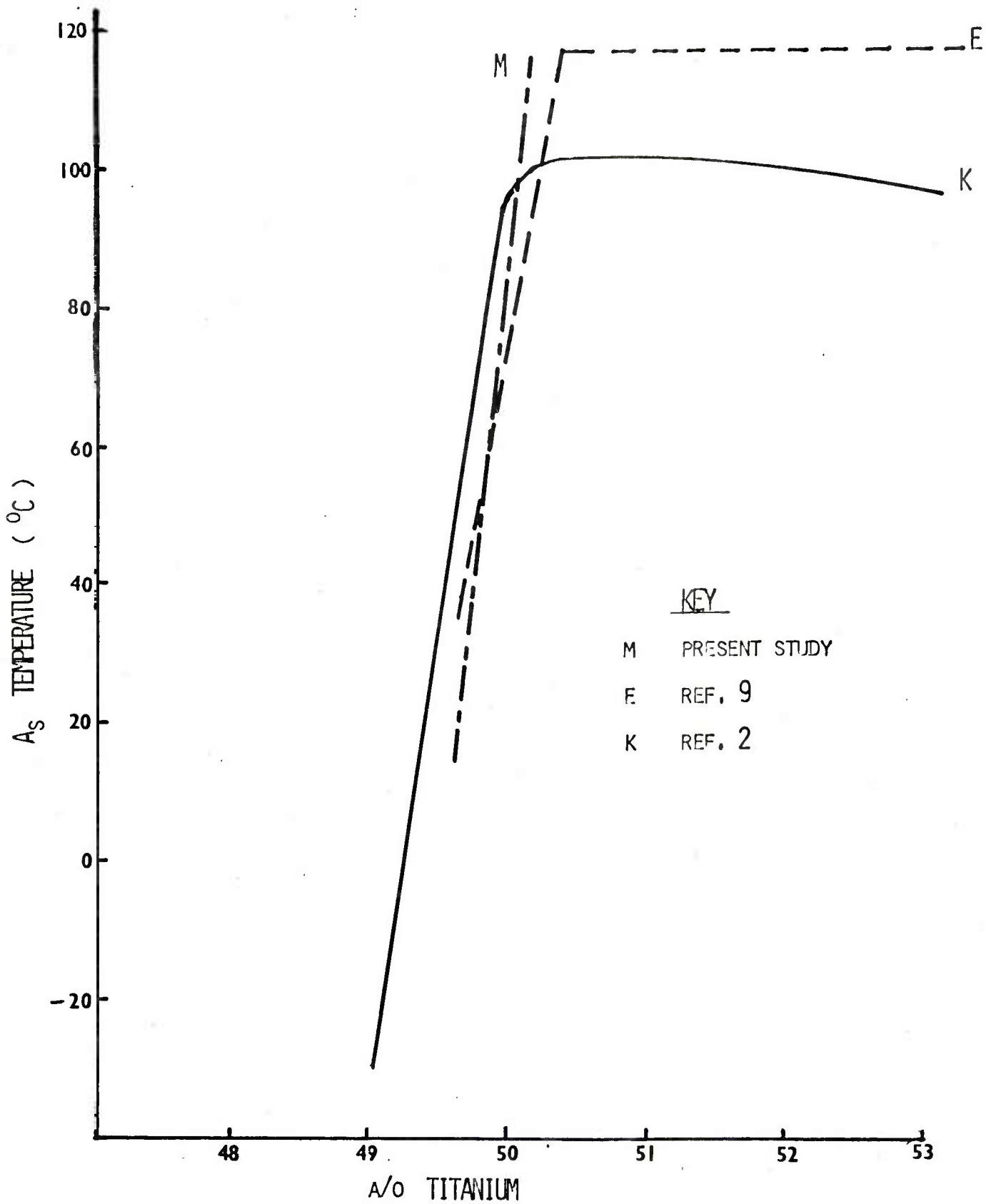


Figure 9

$A_S$  TEMPERATURE vs. ATOMIC PERCENT TITANIUM



# TECHNICAL REPORT INTERNAL DISTRIBUTION LIST

	<u>NO. OF COPIES</u>
COMMANDER	1
CHIEF, DEVELOPMENT ENGINEERING BRANCH	1
ATTN: DRDAR-LCB-DA	1
-DM	1
-DP	1
-DR	1
-DS	1
-DC	1
CHIEF, ENGINEERING SUPPORT BRANCH	1
ATTN: DRDAR-LCB-SE	1
-SA	1
CHIEF, RESEARCH BRANCH	2
ATTN: DRDAR-LCB-RA	1
-RC	1
-RM	1
-RP	1
CHIEF, LWC MORTAR SYS. OFC.	1
ATTN: DRDAR-LCB-M	
CHIEF, IMP. 81MM MORTAR OFC.	1
ATTN: DRDAR-LCB-I	
TECHNICAL LIBRARY	5
ATTN: DRDAR-LCB-TL	
TECHNICAL PUBLICATIONS & EDITING UNIT	2
ATTN: DRDAR-LCB-TL	
DIRECTOR, OPERATIONS DIRECTORATE	1
DIRECTOR, PROCUREMENT DIRECTORATE	1
DIRECTOR, PRODUCT ASSURANCE DIRECTORATE	1

NOTE: PLEASE NOTIFY ASSOC. DIRECTOR, BENET WEAPONS LABORATORY, ATTN:  
DRDAR-LCB-TL, OF ANY REQUIRED CHANGES.

# TECHNICAL REPORT EXTERNAL DISTRIBUTION LIST

	NO. OF COPIES		NO. OF COPIES
ASST SEC OF THE ARMY RESEARCH & DEVELOPMENT ATTN: DEP FOR SCI & TECH THE PENTAGON WASHINGTON, D.C. 20315	1	COMMANDER US ARMY TANK-AUTMV R&D CMD ATTN: TECH LIB - DRDTA-UL MAT LAB - DRDTA-RK WARREN MICHIGAN 48090	1 1
COMMANDER US ARMY MAT DEV & READ. CMD ATTN: DRCDE 5001 EISENHOWER AVE ALEXANDRIA, VA 22333	1	COMMANDER US MILITARY ACADEMY ATTN: CHMN, MECH ENGR DEPT WEST POINT, NY 10996	1
COMMANDER US ARMY ARRADCOM ATTN: DRDAR-LC -ICA (PLASTICS TECH EVAL CEN) -ICE -LCM -ICS -ICW -TSS(STINFO) DOVER, NJ 07801	1 1 1 1 1 2	COMMANDER REDSTONE ARSENAL ATTN: DRSMI-RB -RRS -RSM ALABAMA 35809  COMMANDER ROCK ISLAND ARSENAL ATTN: SARRI-ENM (MAT SCI DIV) ROCK ISLAND, IL 61202	2 1 1 1
COMMANDER US ARMY ARRCOM ATTN: DRSAR-LEP-L ROCK ISLAND ARSENAL ROCK ISLAND, IL 61299	1	COMMANDER HQ, US ARMY AVN SCH ATTN: OFC OF THE LIBRARIAN FT RUCKER, ALABAMA 36362	1
DIRECTOR US Army Ballistic Research Laboratory ATTN: DRDAR-TSB-S (STINFO) ABERDEEN PROVING GROUND, MD 21005	1	COMMANDER US ARMY FGN SCIENCE & TECH CEN ATTN: DRXST-SD 220 7TH STREET, N.E. CHARLOTTESVILLE, VA 22901	1
COMMANDER US ARMY ELECTRONICS CMD ATTN: TECH LIB FT MONMOUTH, NJ 07703	1	COMMANDER US ARMY MATERIALS & MECHANICS RESEARCH CENTER ATTN: TECH LIB - DRXMR-PL WATERTOWN, MASS 02172	2
COMMANDER US ARMY MOBILITY EQUIP R&D CMD ATTN: TECH LIB FT BELVOIR, VA 22060	1		

NOTE: PLEASE NOTIFY COMMANDER, ARRADCOM, ATTN: BENET WEAPONS LABORATORY, DRDAR-ICB-TL, WATERVLIET ARSENAL, WATERVLIET, N.Y. 12189, OF ANY REQUIRED CHANGES.



# TECHNICAL REPORT EXTERNAL DISTRIBUTION LIST (CONT)

	NO. OF COPIES		NO. OF COPIES
COMMANDER US ARMY RESEARCH OFFICE P.O. BOX 12211 RESEARCH TRIANGLE PARK, NC 27709	1	COMMANDER DEFENSE TECHNICAL INFO CENTER ATTN: DTIA-TCA CAMERON STATION ALEXANDRIA, VA 22314	12
COMMANDER US ARMY HARVEY DIAMOND LAB ATTN: TECH LIB 2800 POWDER MILL ROAD ADELPHIA, ME 20783	1	METALS & CERAMICS INFO CEN BATTELLE COLUMBUS LAB 505 KING AVE COLUMBUS, OHIO 43201	1
DIRECTOR US ARMY INDUSTRIAL BASE ENG ACT ATTN: DRXPE-MT ROCK ISLAND, IL 61201	1	MECHANICAL PROPERTIES DATA CTR BATTELLE COLUMBUS LAB 505 KING AVE COLUMBUS, OHIO 43201	1
CHIEF, MATERIALS BRANCH US ARMY R&S GROUP, EUR BOX 65, FPO N.Y. 09510	1	MATERIEL SYSTEMS ANALYSIS ACTV ATTN: DRXSY-MP ABERDEEN PROVING GROUND MARYLAND 21005	1
COMMANDER NAVAL SURFACE WEAPONS CEN ATTN: CHIEF, MAT/SCIENCE DIV DAHLGREN, VA 22448	1		
DIRECTOR US NAVAL RESEARCH LAB ATTN: DIR, MECH DIV CODE 26-27 (DOC LIB) WASHINGTON, D. C. 20375	1 1		
NASA SCIENTIFIC & TECH INFO FAC. P. O. BOX 3757, ATTN: ACQ BR BALTIMORE/WASHINGTON INTL AIRPORT MARYLAND 21240	1		

NOTE: PLEASE NOTIFY COMMANDER, ARRADCOM, ATTN: BENET WEAPONS LABORATORY, DRDAF-ICB-TL, WATERVLIET ARSENAL, WATERVLIET, N.Y. 12189, OF ANY REQUIRED CHANGES.